National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2780

Hard Rock Mine Waste

This Standard Reference Material (SRM) is intended for use in the evaluation of methods and for the calibration of apparati used to determine heavy metals and other elements in hard rock mine waste and materials of a similar matrix. SRM 2780 is composed of material collected from a waste pile of an abandoned mine site near Silverton, CO. A unit consists of approximately 50 g of material of which 90 % passes a 150 µm (No. 100) sieve.

Certified Values and Uncertainties: Certified mass fraction values for 12 elements are listed in Table 1. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST. All values are reported as mass fractions [1], on a dry mass basis (see *Instructions for Drying*), and are based on measurements using a sample mass of at least 250 mg.

Reference Values and Uncertainties: Reference mass fraction values for seven elements are given in Table 2. Reference values are noncertified values that are the best estimate of the true value; however, the values **DO NOT** meet the NIST criteria for certification and are provided with associated uncertainties that may not include all sources of uncertainty. Mass fraction values are on a dry mass basis.

Information Values: Information mass fraction values for 28 elements are provided in Table 3. An information value is considered to be a value that will be of interest and use to the SRM user, but insufficient information is available to assess the uncertainty associated with the value.

Expiration of Certification: The certification of this SRM lot is valid within the measurement uncertainties specified until **31 December 2012**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see *Instruction for Use* section). However, the certification will be nullified if the SRM is contaminated or modified.

Stability: This material is considered to be stable. NIST will monitor this material and will report any substantive changes in certification to the purchaser. Return of the attached registration card will facilitate notification.

The overall direction and coordination of the technical measurements leading to the certification of this SRM were performed by G.C. Turk of the NIST Analytical Chemistry Division.

Analytical measurements from NIST were performed by C.M. Beck II, W.R. Kelly, S.E. Long, J.L. Mann, A.F. Marlow, J.R. Sieber, R.D. Vocke, Jr., and L.L. Yu of the Analytical Chemistry Division.

Statistical consultation was provided by S.D. Leigh and D.D. Leber of the NIST Statistical Engineering Division.

Mine waste sample collection, preparation of the SRM, and contributing chemical analyses were performed by the U.S. Geological Survey (USGS) under the direction of S.A. Wilson. Details are described [4] in USGS Open-File Report 99-370, which is available from USGS Information Services, Box 25286, Federal Center, Denver, CO 80225 (telephone: (303) 202-4210; e-mail: infoservices@usgs.gov).

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by B.S. MacDonald.

Willie E. May, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 31 January 2003

John Rumble, Jr., Chief
Measurement Services Division

Partial financial support for the development of this SRM was provided by the U.S. Environmental Protection Agency (EPA) under the direction of project manager B.A. Schumacher of the EPA National Exposure Research Laboratory, Characterization Research Division, Las Vegas, NV.

INSTRUCTIONS FOR USE

To relate analytical determinations to the certified values on this certificate, a minimum sample mass of 250 mg should be used. Sampling and sample preparation procedures should be designed to avoid material segregation on the basis of particle size. It is recommended to mix the contents of the bottle prior to sampling by turning the bottle end over end for two minutes. The sample should be dried according to the *Instructions for Drying*. If particle size reduction is performed, it is the user's responsibility to prevent contamination or loss of material. This SRM must be stored in an air-conditioned or similar cool and dry environment away from light and fumes.

Instructions for Drying: Analyses should be performed on samples as received; separate samples should be weighed before and after drying to obtain a correction factor for moisture. Samples may be dried in an oven at 107 ºC under clean, dry inert gas, or in a desiccator over fresh anhydrous $Mg(ClO_4)_2$, or by other equivalent method which reaches a constant dry weight.

Table 1. Certified Mass Fractions

Element	Mass Fraction (%)			*k*
Aluminum	8.87	±	0.33	2.3
Calcium	0.195	±	0.020	2.6
Iron	2.784	±	0.080	2.4
Lead	0.577	±	0.041	2.4
Magnesium	0.533	±	0.020	2.8
Potassium	3.38	±	0.26	2.8
Sodium	0.221	±	0.018	2.8
Sulfur	1.263	±	0.042	2.6
Zinc	0.257	±	0.016	2.6

Element	Mass Fraction (mg/kg)			*k*
Arsenic	48.8	±	3.3	2.0
Cadmium	12.10	±	0.24	2.8
Mercury	0.710	±	0.042	2.6

The certified values for mercury and sulfur are the means of results obtained by NIST using isotope dilution mass spectrometry (IDMS). The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined standard uncertainty calculated according to the ISO Guide [2]. The coverage factor, *k*, is determined from the Student's *t*-distribution for the appropriate degrees of freedom to yield 95 % confidence. The certified values and uncertainties for the remaining elements are derived from the results of at least one analysis performed at NIST and independent results from one or more methods provided by the U.S. Geological Survey (USGS) using the approach described by Levenson et al. [3] for combining results for multiple methods. Multiple method results from USGS were first combined to give a single value and uncertainty before being combined with the NIST results. The certified value is an unweighted mean of the results from NIST and USGS. The uncertainty listed with each value is an expanded uncertainty about the mean, $U = ku_c$, with a coverage factor, *k*, determined from the Student's *t*-distribution for the appropriate degrees of freedom to yield 95 % confidence. Each u_c is calculated by combining a between-method variance [3] with a pooled, within-method variance following the ISO/NIST Guide [2]. Analytical methods are listed in Table 4.

Table 2. Reference Mass Fractions

Element	Mass Fraction (%)		k
Titanium	0.699	± 0.019	2.8

Element	Mass Fraction (mg/kg)		k
Barium	993	± 71	2.1
Copper	215.5	± 7.8	2.4
Manganese	462	± 21	2.6
Phosphorus	427	± 40	2.3
Strontium	217	± 18	2.2
Vanadium	268	± 13	2.4

The reference values are based on the results of a single NIST method and one or more USGS methods. Reference values and uncertainties were derived from multiple results in the same manner as was done for the certified values and uncertainties. Analytical methods are listed in Table 4.

Table 3. Information Mass Fractions

Element	Mass Fraction (%)
Silicon	31

Element	Mass Fraction (mg/kg)	Element	Mass Fraction (mg/kg)
Antimony	160	Niobium	18
Cerium	64	Rubidium	175
Cesium	13	Scandium	23
Chromium	44	Selenium	5
Cobalt	2.2	Silver	27
Gallium	26	Tellurium	5
Gold	0.18	Terbium	0.58
Hafnium	4.4	Thallium	5
Holmium	0.84	Thorium	12
Lanthanum	38	Thulium	0.4
Lithium	18	Tungsten	24
Molybdenum	11	Uranium	4
Neodymium	28	Zirconium	176
Nickel	12		

Table 4. Methods Used in Elemental Determinations

Method	Elements Determined
Cold Vapor Isotope Dilution Inductively Coupled Plasma Mass Spectrometry (CV-ID-ICP-MS) at NIST	Hg
Isotope Dilution Thermal Ionization Mass Spectrometry (ID-TIMS) at NIST	S
Standard Additions Inductively Coupled Plasma Mass Spectrometry (ICP-MS) at NIST	As, Cd, Cu, Pb, Zn
Wavelength Dispersive X-ray Fluorescence Spectrometry (WDXRF) at NIST	Al, As, Ba, Ca, Fe, K, Mg, Mn, Na, P, Pb, Sr, Ti, V, Zn, Zr
Wavelength Dispersive X-ray Fluorescence Spectrometry (WDXRF) at USGS	Al, Ca, Fe, K, Mg, Ti
Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) at USGS	Al, Ba, Ca, Cd, Cu, Fe, K, Mg, Mn, Na, P, Pb, Sr, V, Zn
Instrumental Neutron Activation Analysis (INAA) at USGS	As, Ba, Cd, Fe, Na, Zn
Hydride Generation Atomic Absorption Spectrometry (HGAAS) at USGS	As

REFERENCES

[1] Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811, 1995 Ed. (April 1994).

[2] *Guide to the Expression of Uncertainty in Measurement*; SBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NST Technical Note 1297, U.S. Government Printing Office, Washington, DC (1994); available at http://physics.nist.gov/Pubs/.

[3] Levenson, M.S.; Banks, D.L.; Eberhardt, K.R.; Gill, L.M.; Guthrie, W.F.; Liu, H.K.; Vangel, M.G.; Yen, J.H.; Zhang, N.F.; *An Approach to Combining Results from Multiple Methods Motivated by the ISO GUM*; Res. Natl. Inst. Stand. Technol. 105, pp. 571-579 (2000).

[4] Wilson, S.A.; Briggs, P.H.; Brown, Z.A.; Taggart, J.E.; Knight, R.; *Collection, Preparation and Testing of NIST Hard Rock Mine Waste Reference Material SRM 2780*; SGS Open-File Report 99-370, U.S. Geological Survey, Denver, CO (1999).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.